Published in final edited form as:

J Occup Environ Hyg. 2016 June ; 13(6): D93–D98. doi:10.1080/15459624.2016.1153804.

Calibration of high flow rate thoracic-size selective samplers

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Abstract

High flow rate respirable size selective samplers, GK4.126 and FSP10 cyclones, were calibrated for thoracic-size selective sampling in two different laboratories. The National Institute for Occupational Safety and Health (NIOSH) utilized monodisperse ammonium fluorescein particles and scanning electron microscopy to determine the aerodynamic particle size of the monodisperse aerosol. Fluorescein intensity was measured to determine sampling efficiencies of the cyclones. The Health Safety and Laboratory (HSL) utilized a real time particle sizing instrument (Aerodynamic Particle Sizer) and poly-disperse glass sphere particles and particle size distributions between the cyclone and reference sampler were compared. Sampling efficiency of the cyclones were compared to the thoracic convention defined by the American Conference of Governmental Industrial Hygienists (ACGIH)/Comité Européen de Normalisation (CEN)/ International Standards Organization (ISO). The GK4.126 cyclone showed minimum bias compared to the thoracic convention at flow rates of 3.5 l min^{-1} (NIOSH) and 2.7–3.3 l min^{-1} (HSL) and the difference may be from the use of different test systems. In order to collect the most dust and reduce the limit of detection, HSL suggested using the upper end in range (3.3 l min^{-1}). A flow rate of 3.4 l min^{-1} would be a reasonable compromise, pending confirmation in other laboratories. The FSP10 cyclone showed minimum bias at the flow rate of 4.0 l min^{-1} in the NIOSH laboratory test. The high flow rate thoracic-size selective samplers might be used for higher sample mass collection in order to meet analytical limits of quantification.

Keywords

FSP10; GK2.69; GK4.126; sampling efficiency; thoracic-size selective sampler

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Introduction

The size selectivity of respirable-size selective samplers operating at high flow rates (flow rate > 4 l min^{-1}) for respirable crystalline silica (RCS) measurement have been evaluated previously. These provide increased amounts of RCS for more reliable quantitative measurements.[1–3] One high flow rate sampler, the GK2.69 cyclone was developed as a dual use sampler for respirable and thoracic-size selective sampling at flow rates of 4.2 and 1.6 l min^{-1}, respectively.[4] The GK4.162 cyclone, a natural extension of the GK2.69, was recently developed to operate at a higher flow rate, 8.5 l min^{-1} for respirable size selective sampling.[5] Another high flow respirable size selective cyclone, the FSP10, was tested at the National Institute for Occupational Safety and Health (NIOSH) where it showed similar performance to other commonly used respirable size selective samplers.[1–3,6] Like the GK2.69 cyclone, these two high flow rate cyclones might be used for dual fraction size selective sampling to measure the respirable and thoracic-size fractions. Therefore, NIOSH and the Health Safety Laboratory (HSL) carried out work to determine the flow rate at which the cyclone's penetration characteristics most closely agreed with the thoracic-size convention defined by the American Conference of Governmental Industrial Hygienists (ACGIH)[7]/Comité Européen de Normalisation (CEN)[8]/International Standards Organization (ISO).[9]

Methods

Test samplers

The three size selective samplers employed in this study were (1) GK2.69 (BGI by Mesa Labs, Butler, NJ), (2) GK4.126 (Mesa Labs, Butler, NJ), and (3) FSP10 (GSA Messgerätebau GmbH, Ratingen, Germany).

Experiments at NIOSH

The cyclones were tested with six sizes of monodisperse ammonium fluorescein particles generated using a vibrating orifice aerosol generator (VOAG, Model 3450, TSI Inc., Shoreview, MN). This procedure is well documented.[1,10,11] The test cyclone and a thin-walled tube reference sampler were loaded with polyvinyl chloride filters (PVC, GLA-5000, 5 μm pore size, SKC Inc., Eighty Four, PA) and placed horizontally inside the chamber positioned at the same sampling plane. The flow rates of the reference samplers were the same as the test cyclone and the inlet diameter for the reference sampler was calculated in accordance with criteria for calm air sampling[12,13] to ensure minimum sampling bias. The reference sampler was 71-mm long with inlet diameters of 11, 16, and 17 mm for flow rates of 1.6 l min^{-1} (GK2.69), 3.5 l min^{-1} (GK4.126), and 4.0 l min^{-1} (FSP10), respectively. The flow rate required for each cyclone to obtain a sampling efficiency of 50% at approximately 10 μm particle size was initially determined. The sampling efficiencies for other particle sizes were then determined. In order to minimize sampling efficiency error from sampling pump pulsation,[14] sampling flow rates were controlled by mass flow controllers (model CFC 17, Aalborg, Orangeburg, NY) and sampling was conducted between 3 and 6 min depending on the generated particle size. Three repetitions with each cyclone were conducted at each particle size. After sampling, the PVC filters were placed in a 5%

ammonium hydroxide solution to extract the fluorescein and the fluorescein intensity was measured using a luminescence spectrometer (LS50B, Perkins-Elmer, Waltham, MA).

Because the size interval of the APS for particles >8μm is large (>0.6μm), projected area diameter of the monodisperse ammonium fluorescein particles were measured with a field emission scanning electron microscopy (FESEM, Model S-4800-2, Hitachi High Technologies America Inc., Pleasanton, CA). Ammonium fluorescein particles were collected on polycarbonate filters for each particle size and particle equivalent volume diameter (diameter of a sphere of the same volume) was calculated from the projected area diameter measured using the FESEM. From this equivalent volume diameter, an aerodynamic diameter was calculated with the particle's specific gravity (1.35) and dynamic shape factor.[13]

The measured performance data for the cyclone was assessed against the thoracic target convention defined in ACGIH[7]/CEN[8]/ISO,[9] using the bias map approach described in BS EN 13205.[15] The bias between the measured performance curve and the target convention for an array of challenge size distributions was calculated.

Experiments at HSL

HSL just tested the GK4.126 cyclone using an evaluation method consistent with that described in BS EN 13205.[15] The design of the test system was based on that described by Kenny and Lidén[16] used for the measurement of aerosol penetration through cyclone samplers. The approach requires measurements of the aerodynamic size distribution of an aerosol penetrating through the sampler under test and that of the aerosol challenging it. The two size distributions are compared to obtain the penetration characteristics of the sampler.

A powder of ballotini glass beads (Spheriglass 5000, Potters Industries Inc., South Yorkshire, UK) was generated in a calm air chamber using a rotating brush generator (Model RBG 1000, Palas GmbH, Karlsruhe, Germany). The charge level on the aerosol was equalized using an ionizing air blower. This produced an aerosol that was stable with both time and position within the chamber. The particle size distribution of the aerosol was analyzed using an APS (Model 3321, TSI Inc., Shoreview, MN), the calibration of which was checked before testing using traceable polymer microspheres (Duke Scientific Corporation, Fremont, CA) of geometric diameters 3, 5, and 10 μm.

The aerosol was drawn through the cyclone (without filter) and then compared with aerosol drawn through an identical set of tubing, but with no cyclone attached (defined as the challenge or reference aerosol). The dust generator was adjusted to give a concentration of particles (<100 particles cm^{-3}) that resulted in good penetration results, but which was not so high as to create particle coincidence errors within the APS instrument.

The cyclone was characterized for a range of flow rates. Samples of one-minute duration were drawn through the reference line and sampler in turn, allowing a 1-min gap between samples to ensure complete replacement of aerosol in the tubing. In each case three reference and two cyclone samples were taken. Three repeat measurements were made at each flow rate.

The cyclone penetration was calculated as the fraction of average cyclone to average reference particle concentration for each particle size. The particle size at which 50% of the particles penetrated the cyclone (defined as d_{50}, cut off diameter) was then evaluated using curve fitting software.

The measured performance of each cyclone was assessed against the thoracic convention defined in ACGIH[7]/CEN[8]/ISO[9] as described above.

Results

Experiments at NIOSH

Aerodynamic diameters calculated from the FESEM measurements and obtained from the APS measurement are shown in Table 1. The average diameters from the FESEM were significantly larger than those from the APS except at 13 μm (Mann-Whitney Rank Sum Test, SigmaPlot, Systat Software Inc., San Jose, CA). The sampling efficiency curves for GK2.69, GK4.126, and FSP10 cyclones tested with monodisperse ammonium fluorescein particles along with the ACGIH[7]/CEN[8]/ISO[9] thoracic convention are shown in Figure 1. The measured d_{50}s for the GK2.69, GK4.126, and FSP10 cyclones were 9.7 (at 1.6 l min^{-1}), 9.8 (at 3.5 l min^{-1}), and 10.9 (at 4.0 l min^{-1}) μm, respectively. The d_{50}s were calculated from curves fitted (using sigmoid, 3-parameter curve fit) to the measured cyclone sampling efficiencies. Bias maps for the cyclones are shown in Figure 2. The sampling efficiency of the GK2.69 cyclone was measured in the present study to provide assurance that our methodology would give comparable results to a previous calibration study and similar results were observed.[17] The estimated biases for the experimental GK2.69 cyclone performance compared with the ACGIH[7]/CEN[8]/ISO[9] thoracic convention were negative up to 25% while those of the GK4.126 and FSP10 cyclones were positive up to 7 and 11%, respectively for all of the aerosol size distributions.

Experiments at HSL

The cyclone flow rate was checked before and after each test and was found to be within 1% of the target value. Average and standard deviation of the d_{50}s at each flow rate for the GK4.126 are shown in Table 2. Graphs of sampling efficiency for the GK4.126 at different flow rates is shown in Figure 3. Sampler bias at each size distribution and for each flow rate are shown in Figure 4. No large difference in bias (>10%) was found for particle size distributions that had a mass median aerodynamic particle size (MMAD) <20 μm and a geometric standard deviation (GSD) >2.0, irrespective of sampler flow rate.

Discussion

Two different laboratories have tested high flow rate samplers for the measurement of the thoracic-size fraction. The flow rate of the GK4.126 cyclone for size selection was found to be slightly different (2.7–3.3 l min^{-1} {HSL} vs. 3.5 l min^{-1} {NIOSH}) which may be attributable to the difference in test systems (especially differences in the particle sampling methods) and limitation of the APS. For example, the APS can only size particles up to 20 μm, and although the penetration curves appear to cross the x-axis at below 20 μm, the numbers of particles larger than about 14 μm were very low resulting in large measurement errors. This will affect the calculation of sampler bias especially for size distributions with large MMADs and it is a limitation of the test method, which has been noted in previous studies.[18,19] In order to increase the accuracy of measurement up to 20 μm the studies increased the relative concentration of particles larger than 10 μm using a virtual impactor. Maynard et al.[18] observed an apparent increase in penetration above 15 μm without the virtual impactor and a decrease to zero with the impactor in place. The present study did not observe the same apparent increase above 15 μm without using the impactor, possibly due to the different models of APS used in the different studies. Maynard et al.[18] used an APS model 3310 whereas the present study used the later model 3321 which is able to size larger particles more accurately.

The GK4.162 cyclone sampler was found to best agree with the thoracic sampling convention at flow rates between 2.7 and 3.3 l min^{-1} by the HSL. Between these flows, the d_{50} was within 0.3–5% of the target value of 10 μm. In addition, between these flows, the calculated sampler bias was ≤ 10% for more than 85% of the size distributions that it was calculated over (size distributions with mass median aerodynamic diameters (MMAD) between 1 and 30 μm and geometric standard deviations (GSD) between 1.75 and 4). The highest flow-rate value is in good agreement with the value of 3.5 l min^{-1} determined by NIOSH. In addition, if the aerodynamic diameter was underestimated by the APS at HSL, (as observed by NIOSH), the flow rate difference between two laboratories would be even smaller (i.e., the HSL values would be increased slightly). Based on these experiments the GK4.126 appears to agree most closely with the thoracic convention between 3.3 and 3.5 l min^{-1}. Therefore, it is recommended that the GK4.126 should be operated at a flow rate of 3.4 l min^{-1} pending confirmation in other laboratories.

Conclusions

High flow rate GK4.162 and FSP10 cyclones that were initially designed for respirable size selective sampling were calibrated to measure the thoracic-size fraction and were found to conform at flow rates of 3.4 and 4.0 l min^{-1}, respectively. Higher flow rate thoracic samplers will collect more sample for subsequent analysis resulting in an increase in sensitivity making them potentially more useful for the measurement of low concentration aerosols or during short term or task specific sampling. The cyclones should be further investigated for sampling of specific occupational aerosols.

Acknowledgments

Funding

This work was funded by the National Institute for Occupational Safety and Health (NIOSH). Its contents, including any opinions and/or findings/conclusions, are those of the authors alone and do not necessarily represent the official position of the Centers for Disease Control/Prevention, the Agency for Toxic Substances and Disease Registry, or HSE policy.

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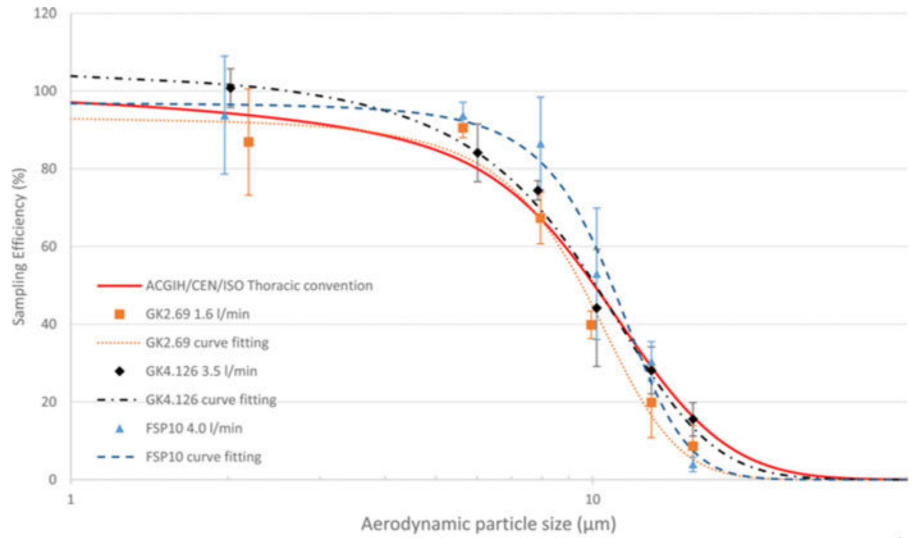

Figure 1.

Average and standard deviation of sampling efficiency of GK2.96 (1.1 l min^{-1}), GK4.126 (4.0 l min^{-1}), and FSP10 (4.0 l min^{-1}) cyclones with monodisperse ammonium fluorescein particles (Figure courtesy of National Institute for Occupational Safety and Health).

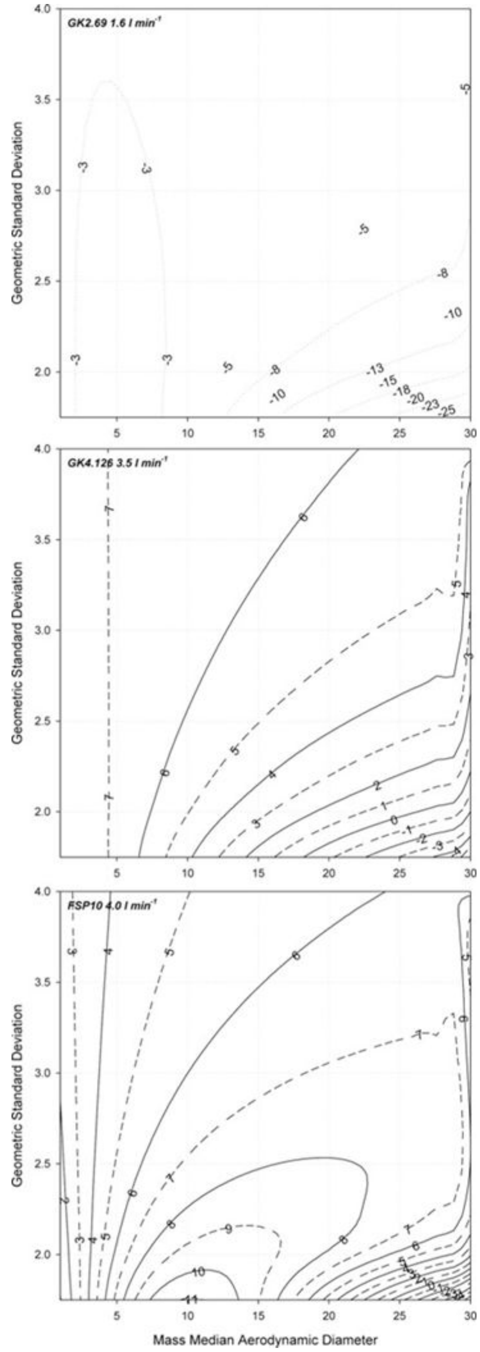

Figure 2.
Bias maps of measured GK2.69 (1.6 l min^{-1}), GK4.126 (3.5 l min^{-1}), and FSP10 (4.0 l min^{-1}) cyclones performance compared to ACGIH/CEN/ISO thoracic convention.

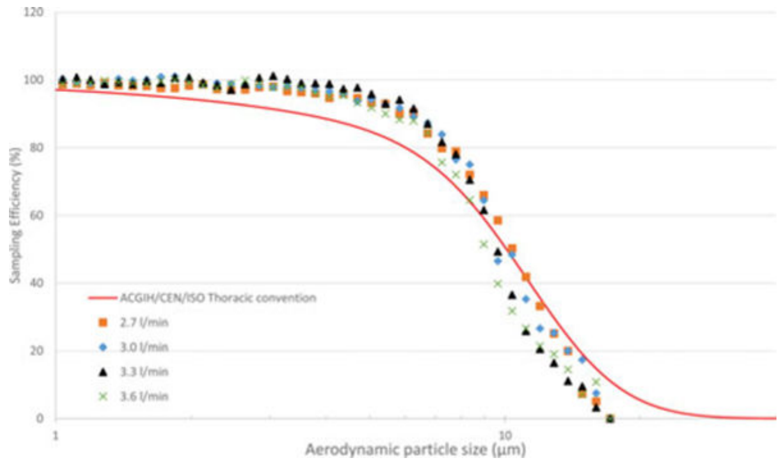

Figure 3.

Average sampling efficiency of GK4.126 cyclone at four different flow rates with polydisperse glass sphere particles (Figure courtesy of Health and Safety Laboratory).

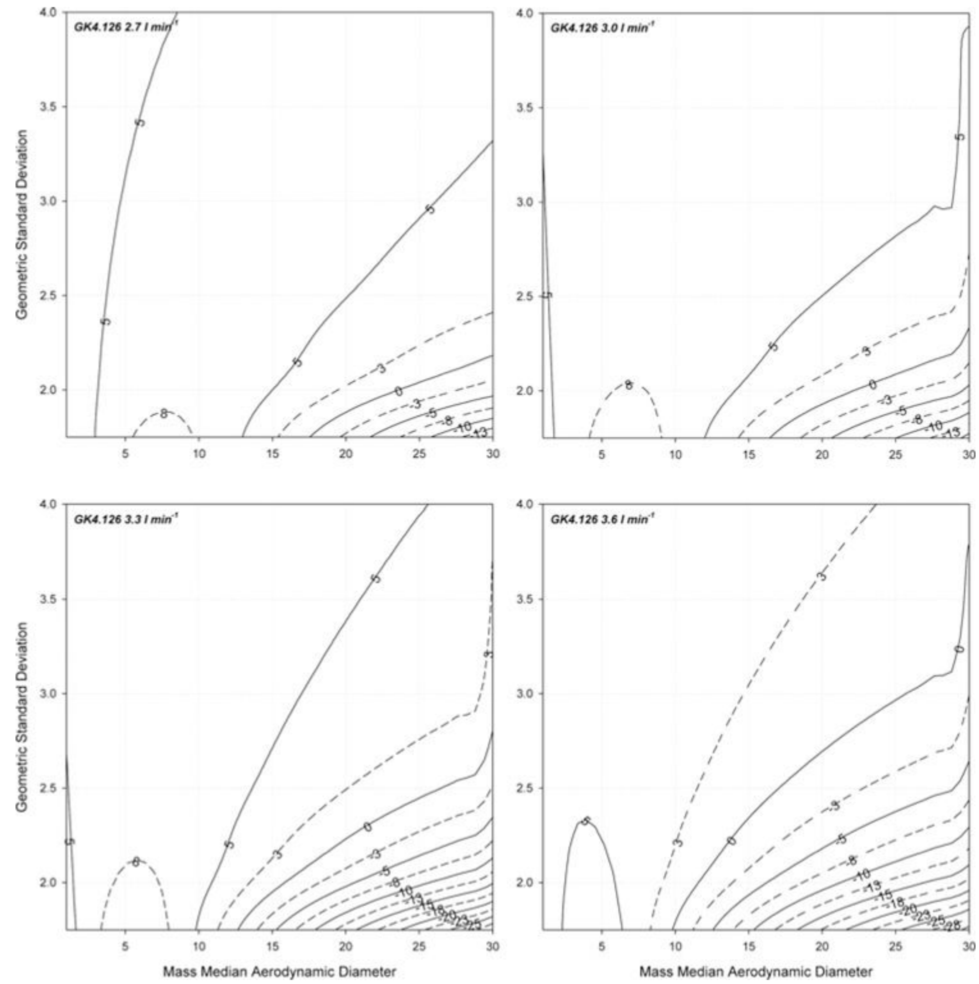

Figure 4.

Bias maps of measured GK4.126 cyclone performance at four different flow rates compared to the ACGIH/CEN/ISO thoracic convention (Figure courtesy of Health and Safety Laboratory).

Table 1

Comparison average and standard deviation of aerodynamic particle diameters between measurements with Aerodynamic Particle Sizer and scanning electron microscope.

Concentration of ammonium flourescein solution (%)	Aerodynamic Particle Sizer measurement	Scanning electron microscope measurement
0.01	2.1 ± 0.1	2.3 ± 0.1
0.5	5.2 ± 0.1	5.6 ± 0.1
1	7.2 ± 0.1	7.9 ± 0.1
2	9.3 ± 0.1	9.9 ± 0.1
4	13.1 ± 0.5	13.0 ± 0.4
8	14.6 ± 0.2	15.6 ± 0.2

Table 2

Cut off diameters of GK4.126 cyclone with different flow rates determined by the Health Safety Laboratory.

Flow rate (l min^{-1})	Cut off diameter (d_{50})					
	Test 1	Test 2	Test 3	Average	Standard deviation	CV[a] (%)
2.7	10.73	9.97	10.81	10.50	0.46	4.4
3.0	9.86	10.15	10.07	10.03	0.15	1.5
3.3	9.55	9.35	9.83	9.58	0.24	2.5
3.6	9.16	9.1	9.14	9.13	0.03	0.3

[a] CV is coefficient of variance.